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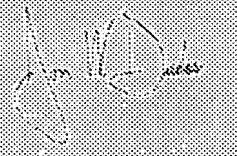
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FILING DATE: *October 21, 2003*  
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Jon W Dudas

Acting Under Secretary of Commerce  
for Intellectual Property  
and Acting Director of the U.S.  
Patent and Trademark Office



## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

17224 U.S. PTO  
Applicant: Ralph A. Petersen  
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Title: BATTERY PASTE MATERIAL  
AND METHOD

Appl. No.: To be determined

Filing Date: 10-21-2003

Examiner: To be determined

Art Unit: To be determined

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I hereby certify that this correspondence is being deposited with the United States Postal Service's "Express Mail Post Office To Addressee" service under 37 C.F.R. § 1.10 on the date indicated below and is addressed to: Commissioner for Patents, PO Box 1450, Alexandria, Virginia 22313-1450.	
<u>EV 227049665 US October 21, 2003</u>	
(Express Mail Label Number) (Date of Deposit)	
<i>Jacqui Banks</i> (Printed Name)	<i>Jacqui Banks</i> (Signature)

**PROVISIONAL PATENT APPLICATION**  
**TRANSMITTAL**

Mail Stop PROVISIONAL PATENT APPLICATION  
Commissioner for Patents  
PO Box 1450  
Alexandria, Virginia 22313-1450

Sir:

Transmitted herewith for filing under 37 C.F.R. § 1.53(c) is the provisional patent application of:

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Applicant claims small entity status under 37 CFR 1.27(c)(1).

Enclosed are:

Specification (15 pages).

Informal drawings (2 sheets, Figures 1-4).

Assignment of the invention to Johnson Controls Technology Company.

Assignment Recordation Cover Sheet.

Small Entity statement(s).

Application Data Sheet (37 CFR 1.76).

The filing fee is calculated below:

	Rate	Fee Totals
Basic Fee	\$160.00	\$160.00
<input type="checkbox"/> Small Entity Fees Apply (subtract 1/2 of above):	=	\$0.00
	TOTAL FILING FEE:	= \$160.00

A check in the amount of \$160.00 to cover the filing fee is enclosed.

The Commissioner is hereby authorized to charge any additional fees which may be required regarding this application under 37 C.F.R. §§ 1.16-1.17, or credit any overpayment, to Deposit Account No. 06-1447. Should no proper payment be enclosed herewith, as by a check being in the wrong amount, unsigned, post-dated, otherwise improper or informal or even entirely missing, the Commissioner is authorized to charge the unpaid amount to Deposit Account No. 06-1447.

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Respectfully submitted,

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**U.S. PROVISIONAL PATENT APPLICATION**

**for**

**BATTERY PASTE MATERIAL AND METHOD**

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## BATTERY PASTE MATERIAL AND METHOD

### FIELD

[0001] The present inventions relate generally to the field of batteries (e.g., lead-acid batteries such as automotive starting, lighting, and ignition batteries; industrial batteries, commercial batteries, and marine batteries). More specifically, the present inventions relate to materials for use in active materials used in batteries and methods of making such materials.

### BACKGROUND

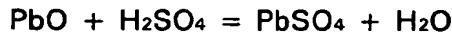
[0002] In producing lead-acid batteries (e.g., lead-acid starting, lighting, and ignition ("SLI") batteries for vehicles), positive and negative plates or grids are produced. Such plates or grids are made of lead or a lead alloy, and include a plurality of wires coupled to a plurality of nodes (e.g., a battery plate may include a frame comprising four sides with a lug or current collector extending from one of the sides and a network of wires or grid elements interconnected with a plurality of nodes).

[0003] At least a portion of the positive grids or plates have a material (e.g., a paste) applied thereto. The paste typically comprises lead oxide (PbO). The active material may also include one or both of tetrabasic lead sulfate ( $4\text{PbO}\bullet\text{PbSO}_4$ ) (often abbreviated as "4BS") and tribasic lead sulfate ( $3\text{PbO}\bullet\text{PbSO}_4\bullet\text{H}_2\text{O}$ ) (often abbreviated as "3BS"). According to an exemplary embodiment, an active material may comprise approximately 40% PbO and 60% 4BS. According to other exemplary embodiments, the active material may have a different composition (e.g., the active material may include between approximately 10% and 100% 4BS, etc.). The tetrabasic lead sulfate and tribasic lead sulfate may be

provided in the form of individual crystals that are mixed into the lead oxide paste material. According to an exemplary embodiment, tetrabasic lead sulfate and tribasic lead sulfate may be provided by adding acid to a paste mix under appropriate mixing and plate curing conditions.

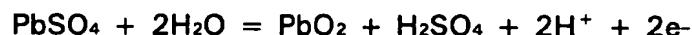
[0004] The positive plates including paste are cured or dried to remove excess liquid in the paste and assembled into a battery (e.g., positive and negative plates are provided with a separator between them in a battery container, after which acid (e.g., sulfuric acid) is introduced into the battery). During curing, the tetrabasic lead sulfate and/or tribasic lead sulfate crystals grow or increase in size.

[0005] During battery formation (e.g., providing an initial charge to the battery), the components of the paste are converted to an active material such as lead dioxide ( $PbO_2$ ) on the positive plates and sponge lead (Pb) on the negative plates. According to an exemplary embodiment, a sulfation reaction proceeds as acid is added to the battery according to the formula:

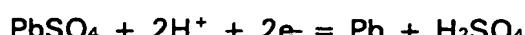


[0006] During formation, according to an exemplary embodiment, the reactions at the positive and negative plates proceeds according to the following formulae:

Positive Plate



Negative Plate



Overall Reaction

[0007] Cured positive plates containing tetrabasic lead sulfate as a component of the paste applied thereto may provide improved deep discharge cycle life as compared to cured positive plates utilizing tribasic lead sulfate as a component in the paste. B. Culpin has provided a review of 4BS positive plate chemistry and its benefits in *J. Power Sources*, 25, p. 305-311 (1989).

[0008] Less well noted in the public literature is the improved discharge capacity of positive plates containing tetrabasic lead sulfate compared to tribasic lead sulfate. For example, batteries produced using tetrabasic lead sulfate positive plate technology have been shown to yield up to approximately 20% improvements in reserve capacity (where reserve capacity is defined as the number of minutes at a 25 ampere discharge at 80°F until a battery voltage is reduced to 10.5 volts). The tetrabasic lead sulfate plate chemistry was produced by subjecting freshly pasted and flash dried positive plates to 2 hours of atmospheric pressure steam, followed by the normal Chemset curing process of 16 hr at 49°C and a humidity of greater than 70%.

[0009] Tetrabasic lead sulfate is conventionally provided having a crystal size of between approximately 10 and 20 micrometers. One difficulty with using such tetrabasic lead sulfate is that the crystal size may not be optimum for allowing conversion of the paste materials into lead dioxide during the formation process. Another difficulty is that the use of such tetrabasic lead sulfate requires that the positive plates undergo a high temperature steam cure for approximately 1 hour or more.

[0010] The plates produced using this method included cured (e.g., before battery formation) tetrabasic lead sulfate crystals having sizes greater than 10 microns in thickness. One detrimental effect of the use of such crystals was that the plates exhibited incomplete formation (i.e., not all tetrabasic lead sulfate was converted to lead dioxide active material). Accordingly, batteries produced with such plates required follow-up boost charging to complete the formation process. The large crystals, coupled with the incomplete formations, also produced warpage of the formed positive plates. Such difficulties in the use of tetrabasic lead sulfate chemistry may render the use of such chemistry undesirable.

[0011] Another difficulty in utilizing tetrabasic lead sulfate chemistry is that the paste mixing process and/or the plate curing step must be performed at elevated temperatures of at least 70°C and more typically exceeding 80°C. Such elevated temperatures may not be desirable for such manufacturing processes.

[0012] There is thus a need to provide an improved method for producing tetrabasic lead sulfate materials for use in battery paste. There is also a need to provide a battery paste having tetrabasic lead sulfate with an optimum crystal size to enable relatively efficient conversion of the tetrabasic lead sulfate into lead dioxide active material. There is also a need to provide a relatively efficient and cost-effective method of producing battery paste for use in lead-acid batteries. There is also a need to provide a method for producing materials for use in battery paste that decrease the material requirements for production of a battery without sacrificing battery performance or cycle life and without reducing manufacturing efficiency. These and other needs may be met by one or more of the preferred and exemplary embodiments described herein.

**OUTLINE OF BASIC AND OTHER ADVANTAGEOUS FEATURES**

**[0013]** It would be desirable to provide a process for manufacturing or producing a material of a type disclosed in the present application that includes any one or more of these or other advantageous features:

1. A method for producing or manufacturing battery plates utilizing tetrabasic lead sulfate paste chemistry that utilizes lower temperatures than conventional methods.
2. A method that provides optimum sized tetrabasic lead sulfate crystals in a battery paste.
3. A method that improves conversion of tetrabasic lead sulfate crystals during battery formation.
4. A method that utilizes tetrabasic lead sulfate plate chemistry to reduce active material paste weights without degrading battery performance or cycle life and without significantly increasing manufacturing costs.
5. A relatively cost-effective and efficient method for manufacturing battery plates utilizing tetrabasic lead sulfate paste chemistry.
6. A relatively simple, robust, and cost effective process for making cured lead acid battery plates with high percent conversion to optimally sized (2-5 micron thick) tetrabasic lead sulfate, that can be relatively efficiently converted into lead dioxide active material during the battery formation process.

7. A method of providing active material for a positive battery plate using conventional low temperature curing while utilizing a battery paste comprising tetrabasic lead sulfate.

8. A method that utilizes relatively small seed crystals of tetrabasic lead sulfate to produce smaller crystals of tetrabasic lead sulfate after a curing operation than possible using conventional methods.

9. An active material that has a higher percentage of tetrabasic lead sulfate converted to lead dioxide during a battery formation process than may be obtained using conventional manufacturing methods.

#### BRIEF DESCRIPTION OF THE DRAWINGS

[0014] FIGURE 1 shows a 2000X magnification scanning electron micrograph of a conventional positive plate utilizing a tribasic lead sulfate chemistry (i.e., without the use of tetrabasic lead sulfate) which had been cured at a low temperature of 46°C for 16 hrs at 95% humidity.

[0015] FIGURE 2 shows a 2000X magnification scanning electron micrograph of a positive plate which had been cured under the same low temperature conditions as for the plate shown in FIGURE 1, but which utilized a paste mix with 1 wt% of a tetrabasic lead sulfate seed crystal additive.

[0016] FIGURE 3 shows a 2000X magnification scanning electron micrograph of a positive plate utilizing tetrabasic lead sulfate having a larger particle size than that used to produce the plates shown in FIGURE 2 (e.g., without the use of tetrabasic lead sulfate seeds).

[0017] FIGURE 4 is a graph illustrating a theoretical quantitative prediction of percent tetrabasic lead sulfate conversion in low temperature cured plates versus seed crystal diameter.

DESCRIPTION OF THE PREFERRED AND EXEMPLARY EMBODIMENTS

[0018] The present inventions relate to a process or method of manufacturing positive plates or grids utilizing tetrabasic lead sulfate as a component of the battery paste (e.g., along with PbO) that provides a savings in positive plate materials (e.g., between 4 and 8%) with little or no loss in lead acid battery performance or cycle life and little or no decline in manufacturing productivity.

[0019] According to an exemplary embodiment, the process allows for paste mixing and curing temperatures of less than 60°C and 46°C to be used, respectively. Such temperatures are significantly lower than temperatures used for conventional tetrabasic lead sulfate plate chemistry manufacturing processes, which may range from approximately 70° to 80° or higher.

[0020] According to an exemplary embodiment, finely ground tetrabasic lead sulfate particles at a loading level of 1 wt% are added to leady oxide in an otherwise standard paste mixing process. According to an exemplary embodiment, the particles have an average particle diameter of approximately 1 micron. According to other exemplary embodiments, the particles may have a different average particle diameter (e.g., 2 microns or greater.

[0021] Following curing of the battery paste at a relatively low temperature, the particles will grow through nucleation and grain growth to sizes smaller than possible using conventional high temperature curing (e.g., between approximately 2 and 5 microns thick, and preferably approximately 3 microns thick). Following the curing step which causes growth of the tetrabasic lead sulfate crystals, the tetrabasic lead sulfate crystals comprise between approximately 50 and 60% by weight of the

cured paste. According to other exemplary embodiments, a higher or lower acid content in the pastes may be used to obtain levels of tetrabasic lead sulfate between approximately 10% and 100% by weight of the cured plate. According to still other exemplary embodiments, the total weight of tetrabasic lead sulfate may also vary based on the amount of tetrabasic lead sulfate particles utilized.

[0022] One advantageous feature of utilizing relatively finely ground tetrabasic lead sulfate particles or "seed crystals" is that greater than approximately 90% conversion of all PbSO<sub>4</sub> may be converted into tetrabasic lead sulfate. No further steam curing or other curing process is required, such as the steam curing process that may be used with conventional tetrabasic lead sulfate production methods.

[0023] The tetrabasic lead sulfate particles or "seed crystals" catalyze the full conversion of all tribasic lead sulfate chemistry into tetrabasic lead sulfate at a curing temperature of between approximately 46° and 48°C, provided that the humidity is maintained at approximately 95%. According to other exemplary embodiments, the humidity may be maintained at a different level (e.g., between approximately 80 and 100%. One advantageous feature of utilizing such temperatures is that lower manufacturing temperatures take less energy and avoid the higher costs of warpage resistant fiber filled plastic stacking boards to hold the plates during the curing process. Further, high paste mixing processes may require more expensive process equipment (e.g., vacuum-cooled paste mixers).

[0024] One advantageous feature of using small tetrabasic lead sulfate seed crystal sizes is that the required amount of tetrabasic lead sulfate is reduced, which thus reduces the cost of this paste mix additive. According to an exemplary embodiment, each tetrabasic lead sulfate seed

crystal develops into a single cured tetrabasic lead sulfate crystal. The greater the number of seed crystals, the greater the number of cured crystals. Because there are a greater number of seed crystals, the final cured crystals have a smaller size than those produced using conventional processes (e.g., the growth of each of the seed crystals into the larger, cured crystals is constrained due to the number of seed crystals provided). The relatively small cured crystal sizes may be produced regardless of curing temperature.

[0025] The tetrabasic lead sulfate particles are produced by jet milling larger particles of tetrabasic lead sulfate to obtain an average particle size of between approximately 1 and 2 microns. According to an exemplary embodiment, a Fluid Energy Aljet Model 8 Micro-Jet Grinding System (manufactured by Fluid Energy Aljet of Telford, Pennsylvania) may be utilized to produce tetrabasic lead sulfate seed crystals or particles having reduced sizes (e.g., between approximately 1 and 2 microns). According to other exemplary embodiments, other types of jet mill equipment may be used. According to other exemplary embodiments, other methods of producing tetrabasic lead sulfate particles having particle sizes smaller than those conventionally used may also be utilized. According to still other exemplary embodiments, the particle size of the tetrabasic lead sulfate particles may be less than 1 micron or greater than 2 microns (e.g., between approximately 2 and 5 microns).

[0026] According to other exemplary embodiments, the average particle size may differ. For example, the average particle size and loading levels may vary to optimize the conversion of tetrabasic lead sulfate to lead dioxide during the formation process. According to one embodiment, the particle size of the tetrabasic lead sulfate particles may range between approximately 2 and 5 microns. According to another exemplary

embodiment, the tetrabasic lead sulfate particles may be provided with a plurality of particle sizes (e.g., approximately 10% of the tetrabasic lead sulfate particles have average particle sizes of between approximately 10 and 20 microns, and 90% of the tetrabasic lead sulfate particles have a particle size of approximately 1 micron). The particular mixture of particle sizes may, of course, vary according to various other exemplary embodiments. According to another exemplary embodiment, the amount of loading of the paste with tetrabasic lead sulfate seed crystals may range between approximately 0.5% and 10.0% by weight. Other loading amounts may also be used according to other exemplary embodiments.

[0027] One advantageous feature of the use of tetrabasic lead sulfate particles having reduced sizes is that the tetrabasic lead sulfate crystals result in a cured tetrabasic lead sulfate crystal size that is small enough to provide relatively efficient conversion to lead dioxide positive active material in the first charge of the lead acid battery (commonly referred to as the formation process).

[0028] FIGURE 1 shows a 2000X magnification scanning electron micrograph of a conventional positive plate utilizing a tribasic lead sulfate chemistry (i.e., without the use of tetrabasic lead sulfate) which had been cured at a low temperature of 46°C for 16 hrs at 95% humidity. The small crystalline structure illustrated in the micrograph is characteristic of conventional tribasic lead sulfate chemistry, as was confirmed by x-ray diffraction and thermal gravimetric analysis (J. Materials Science Letters, Vol. 11, pp 369-372 (1992).

[0029] In contrast, FIGURE 2 shows a scanning electron micrograph at the same 2000X magnification of a plate which had been cured under the same low temperature conditions as for the plate shown in FIGURE 1, but which utilized a paste mix with 1 wt% of a tetrabasic lead sulfate seed

crystal additive. The use of tetrabasic lead sulfate crystals according to an exemplary embodiment provides larger 2 - 3 micron thick crystals. Such cured crystal size is desirable since such crystals are optimally sized to convert to lead dioxide during the battery formation process, while at the same time yielding life and performance improvements over tribasic lead sulfate plate chemistry. X-ray diffraction and thermal gravimetric analyses confirmed that more than 90% of the PbSO<sub>4</sub> present in the plate had been converted into the tetrabasic lead sulfate crystalline form.

[0030] FIGURE 3 shows a 2000X scanning electron micrograph of a plate utilizing tetrabasic lead sulfate having a larger particle size than that used to produce the plates shown in FIGURE 2 (e.g., the tetrabasic lead sulfate particles were made without milled tetrabasic lead sulfate seeds). The plates were steam cured at a temperature of approximately 100°C. The much larger, approximately 10 micron thick tetrabasic lead sulfate are more difficult to convert into lead dioxide positive plate active material during the subsequent battery formation processes. Such plates also show a greater tendency toward warpage during the formation process.

[0031] Use of tetrabasic lead sulfate "seed crystals" of nominal 1-2 micron particle size provides a relatively simple and robust process which assures that the proper size and amount of tetrabasic lead sulfate seed material is in the plate during the subsequent, critical plate curing step.

[0032] The degree of conversion of PbSO<sub>4</sub> into the desired tetrabasic lead sulfate chemistry is also critically controlled by tetrabasic lead sulfate seed crystal particle size at the relatively low curing temperatures which would not otherwise create more tetrabasic lead sulfate crystals during curing. A theoretical quantitative prediction of percent tetrabasic lead sulfate conversion in low temperature cured plates versus seed crystal diameter is shown in FIGURE 4. One assumption forming the basis of

**FIGURE 4** is that low temperature cured tetrabasic lead sulfate crystals cannot grow larger than about 3 microns thick and 30 microns long. The number of these cured crystals determines the percent conversion to tetrabasic lead sulfate in the cured plates. Increasing the number of tetrabasic lead sulfate seed crystals per unit weight of additive via particle size reduction, increases the percent conversion of tetrabasic lead sulfate in the cured plate by creating greater number of nucleation sites to create greater numbers of cured tetrabasic lead sulfate crystals.

**[0033]** FIGURE 4 shows that seed crystal diameters need to be no larger than about 2 microns in diameter to assure full conversion to tetrabasic lead sulfate crystals in the curing process. Still smaller seed crystal sizes would more robustly ensure full conversion to tetrabasic lead sulfate at low curing temperatures and could enable smaller amount of seed crystal additive to reduce process costs.

**[0034]** The paste material utilizing tetrabasic lead sulfate seed crystals yields improvements over conventional tetrabasic lead sulfate plate production by circumventing the need for an additional high temperature steam curing process. The method also generates optimally-sized post-cure tetrabasic lead sulfate crystals that are more efficiently converted to lead dioxide than possible using conventional tetrabasic lead sulfate plate production methods. The use of such seed crystals advantageously retains the benefits of tetrabasic lead sulfate plate chemistry such as a 5-15% increase in positive plate material utilization, improved discharge capacity stability during repetitive reserve capacity testing, and improved deep discharge cycle life.

**[0035]** The following nonexclusive example illustrates features of the present inventions:

Example

[0036] Greater than 90 wt% purity tetrabasic lead sulfate (tribasic lead sulfate contaminant) was prepared in 60 lb lots in 50 gallons of hot aqueous slurries according to a procedure described by Biagetti and Weeks in the September 1970 issue of the Bell System Technical Journal. The dried material was then shipped to Fluid Energy Aljet (Telford, PA) for jet mill grinding to average volume based particle sizes of 1 micron with a nominal standard deviation of 1 microns. Laser based particle size analyzers were used to quantitate all tetrabasic lead sulfate seed particle sizes.

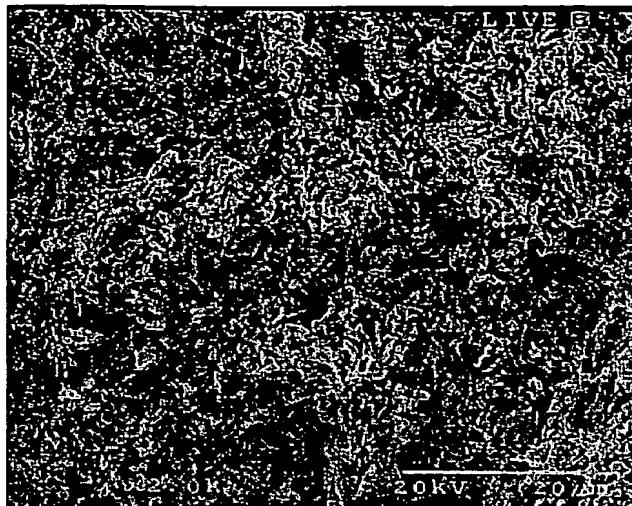
[0037] The tetrabasic lead sulfate seed particles were added to a 2400 lb paste mix of conventional leady oxide to achieve a desired 1 wt% loading level (i.e., 24 lbs. of lead sulfate seeds were added to the mix). Normal state of the art mixing was then conducted via water additions, followed by the appropriate amount of 1.325 specific gravity sulfuric acid addition over a nominal 10 minute period to yield nominal peak mix temperatures of 60°C.

[0038] Machine pasted plates were then flash dried to a nominal moisture content of 10% and then subjected to 16 hours of curing at 46°C and 95% humidity. The plates were then dried for a nominal 30 hours at 60°C at low humidities not to exceed 50%. Conventional battery assembly and formations followed to make the test batteries. Battery Council International (BCI) testing procedures and equipment were used to conduct performance and life testing of all batteries.

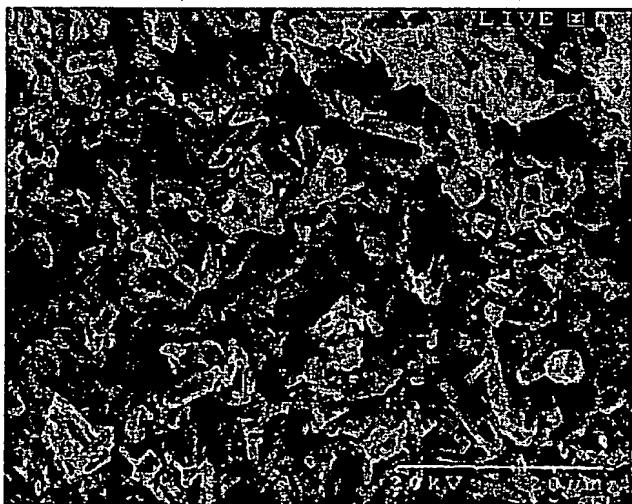
[0039] X-ray diffraction was used to confirm all tribasic lead sulfate and tetrabasic lead sulfate cured plate chemistries, while thermal gravimetric analysis was coupled with chemical sulfate analyses to quantitate these

species according to the procedure described in the Journal of Material Sciences Letters, Vol 11, pp 369-372 (1992).

[0040] It is important to note that the preferred and other exemplary embodiments are illustrative only. Although only a few embodiments of the present inventions have been described in detail in this disclosure, those skilled in the art who review this disclosure will readily appreciate that many modifications are possible (e.g., variations in sizes, dimensions, structures, shapes and proportions of the various elements, values of parameters, etc.) without materially departing from the novel teachings and advantages of the subject matter recited herein. Other substitutions, modifications, changes and omissions may be made in the design, process parameters, material properties, operating conditions and other features of the preferred and other exemplary embodiments without departing from the scope of the present inventions.



**FIGURE 1:** SEM of Conventional Low Temperature (46°C) Cured Plate Made without 4BS Seeds: 2000X Magnification : X-ray diffraction and thermal gravimetric analysis confirmed that all sulfate is present in the form of 3BS.



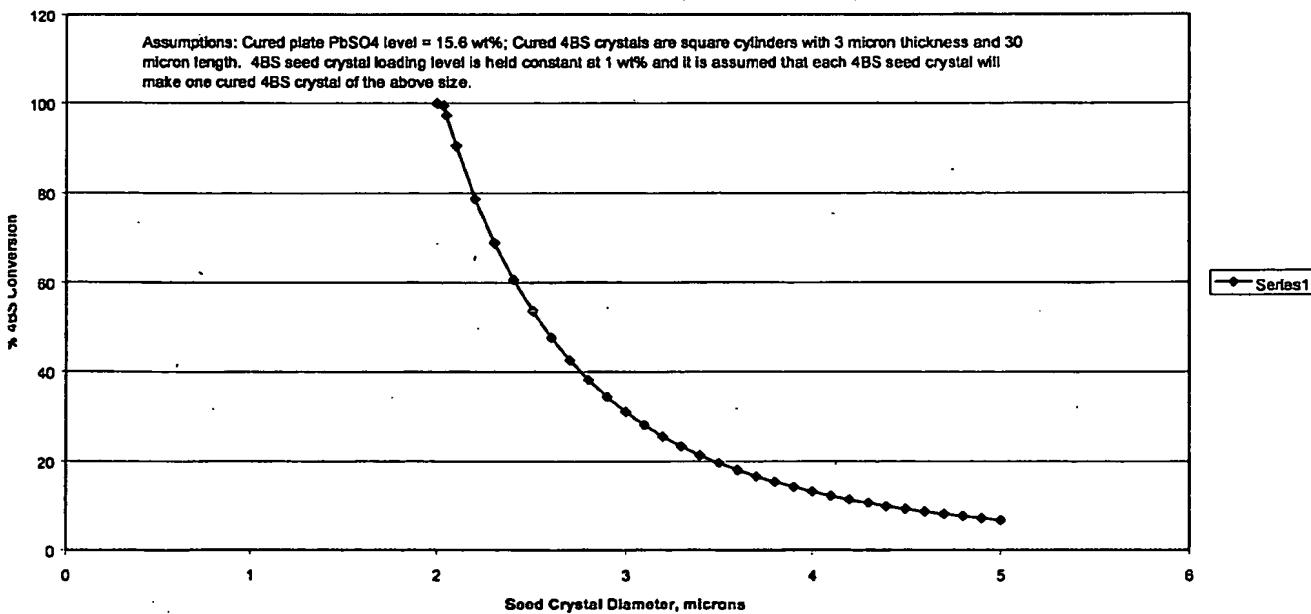
**FIGURE 2:** SEM of Low Temperature Cured (46°C) Plate Made with 4BS Seeds Process to Produce Optimally Sized (2-3 micron thick) 4BS ( $4\text{PbO} \cdot \text{PbSO}_4$ ) crystals,: 2000X Magnification : X-ray diffraction and thermal gravimetric analysis confirmed that greater than 90% of all sulfate is present in the form of 4BS



**FIGURE 3: SEM of High Temperature Steam Cured (100C) Plate Made without 4BS Seeds.**  
**2000X Magnification :** The large 10 micron thick 4BS crystals cause significant inefficiencies during the subsequent formation processing into  $\text{PbO}_2$  positive plate active material. X-ray diffraction and thermal gravimetric analysis confirmed that greater than 90% of all sulfate is present in the form of 4BS.

**FIGURE 4: Theoretical Prediction of Percent 4BS Conversion at Low Temperatures versus Seed Crystal Diameter**

Theoretical % 4BS Conversion vs 4BS Seed Crystal Diameter in Microns



001.1464260.1

## Application Data Sheet

### Application Information

**Application Type::** Provisional  
**Subject Matter::** Utility  
**Suggested classification::**  
**Suggested Group Art Unit::**  
**CD-ROM or CD-R?::** None  
**Computer Readable Form (CRF)?::** No  
**Title::** BATTERY PASTE MATERIAL AND  
METHOD  
**Attorney Docket Number::** 054821-0444  
**Request for Early Publication?::** No  
**Request for Non-Publication?::** No  
**Suggested Drawing Figure::** Not Applicable  
**Total Drawing Sheets::** 2  
**Small Entity?::** No  
**Petition included?::** No  
**Secrecy Order in Parent Appl.?::** No

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Primary	47,959	M. REED STAHELI
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#### Domestic Priority Information

<b>Application::</b>	<b>Continuity Type::</b>	<b>Parent Application::</b>	<b>Parent Filing Date::</b>

### **Foreign Priority Information**

<b>Country::</b>	<b>Application number::</b>	<b>Filing Date::</b>	<b>Priority Claimed::</b>

### **Assignee Information**

**Assignee name::**

Johnson Controls Technology Company

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Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)



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